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Description of CH680292

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The present invention concerns a method to the continuous production of sulphonyl chloride isocyanate, which is shortened in the following with CSI, by conversion of sulphur trioxide and cyanogen chloride (ClCN) in a molar ratio from 1,1 to 1,7:1 at temperatures from 30 to 160 DEG C as well as a plant to the lead-through of the method according to invention.

Of the EP-A 0,294,613 is well-known to make sulphonyl chloride isocyanate by conversion of cyanogen chloride and sulphur trioxide in the molar ratio 1:1 at temperatures from 100 to 200 DEG C. In this method gaseous cyanogen chloride and sulphur trioxide are introduced into the gaseous phase of a reaction zone filled with chloresulfonylisocyanathaltigen fluid and the gaseous phase diverting from the reaction zone is fractionated distilled. The raw CSI resulting thereby is then introduced into the sump of a second distillation column, distilled fractionated, whereby after cooling of the gaseous phase absorbing from the distillation zone liquid sulphonyl chloride isocyanate results. Both the conversion of the reactants and the distillations are accomplished under normal print and on normal atmospheric oxygen conditions.

Disadvantages of this method are:

1. With a molar ratio of 1:1, is the formation of by-products from parallel reactions, as the education makes 2,6-Dichlor-1,4,3,5-oxathiadiazin-4,4-dioxid more difficult (DOD) favoured by Pyrosulfurylchloridisocyanat (psi), and the handling of the process.
 2. The conversion of cyanogen chloride and sulphur trioxide in the gaseous phase leads, under which easily sublimating, solid by-product (2,6-Dichlor-1,4,3,5-oxathiadiazin-4,4-dioxid) which
- ▲ topclogs reaction conditions, to a with difficulty decomposable, the distillation columns.

The task of the invention consisted of switching the disadvantages off from the well-known method and an industrial method to the continuous production of sulphonyl chloride isocyanate of cyanogen chloride and sulphur trioxide developing as well as a plant to the lead-through of the method.

The task could be achieved according to invention with a method in accordance with claim 1 and with a plant in accordance with claim 7.

Gaseous sulphur trioxide and gaseous cyanogen chloride are preferably introduced in a molar ratio from 1,1 to 1,7:1, of, 1.3 to 1,5:1, in with more chlorsulfonylisocyanathaltiger, at temperatures between 30 and 160 DEG C held, fluid filled raw CSI column (6).

The heat of reaction developing thereby is used for the operation of the distillation column. Above the vapors withdrawing from the raw CSI column (6) are then cooled down in a first cooling zone downstream on a temperature under 110 DEG C, preferably on a temperature of under 95 DEG C, and condensed. The raw CSI resulting thereby is led back partially as flyback into the raw CSI column (6), while the other part is transferred of a second distillation column downstream into the lift zone and between lift zone and drift zone is distilled. The gaseous sulphonyl chloride isocyanate diverting thereby is then taken between the lift zone and drift zone and condensed in a cooling zone. The whole method is accomplished under inert gas, preferably under easy N₂-Druck up to 0,5 bar positive pressure.

The vapors withdrawing from the second distillation column are cooled down in a cooling zone downstream on temperatures from 60 to 100 DEG C, preferably on temperatures from 85 to 100 DEG C, and partial condensed, whereby the condensate is led back as return flow into the second distillation column.

The not condensed vapors from the cooling zone downstream of the second distillation column are continued to liquefy in a further cooling zone downstream under 50 DEG C, introduced preferably between 35 and 50 DEG C, and the condensate into the reaction zone.

The not condensed vapors from the first cooling zone downstream are liquefied just as in a further cooling zone downstream under 50 DEG C, preferably between 35 and 50 DEG C, and the condensate into the reaction zone is introduced.

The gases, which do not condense even under these conditions, preferably leave the system.

The CSI haltige fluid from the lower part of the second distillation column can be led back into the reaction zone. The CSI haltigen by-products of the reaction zone can flow off over a discharge system for disposal.

Preferably the gaseous sulphur trioxide from an oleum distillation zone is introduced into the reaction zone. The gaseous cyanogen chloride is preferably introduced from a chlorine cyanide vaporizer, by a drying agent bed, appropriately by a molecular sieve dryer, into the reaction zone.

The plant to the lead-through of the method according to invention is likewise a component of the invention and consists of a drying agent container 3, a CSI generator 5, a raw CSI column 6, a parliamentary group divisor 8, and the raw CSI radiator 7, whereby the drying agent container 3 on the one hand connected by a first wire 2 is led with the ClCN vaporizer 1 and on the other hand across a second wire 4 to the CSI generator 5 under the liquid level and whereby on the CSI generator 5 the raw CSI column 6, reaction divisor 8 and the raw CSI radiator 7 is put on to which; an oleum distillation zone 10 whereby the upper range of the oleum distillation zone 10 with the CSI generator 5 by a third wire 11 is connected; a pure CSI Destillationsblase 12, a pure CSI column 13, a lower pure CSI Fraktionsteiler 14, an upper parliamentary group divisor 16 and a radiator 15, whereby on the distillation blister 12, which pure CSI column 13, which both parliamentary group divisors 14, 16, and the radiator 15 is put on, and whereby the upper range of the pure CSI column 13 with the parliamentary group divisor 8 by a fourth wire 9 is connected and whereby a fifth wire 18 connects the pure CSI Destillationsblase 12 with the CSI generator 5 and whereby the lower pure CSI Fraktionsteiler 14 below the intake line 9 is attached and whereby the parliamentary group divisor 14 a product line 17 goes off; a radiator 19, a parliamentary group divisor 20 and a pressure retaining valve 21 whereby the upper range of the radiator 19 by a sixth wire 24 with the upper range of the raw CSI radiator 7 and by a sieved wire 25 with the upper range of the radiator 15 and whereby the radiator 19, which is flowmoderately connected fourth parliamentary group divisors 20 and the first pressure retaining valve 21 with one another, is connected and whereby the fourth parliamentary group divisor 20 with the CSI generator 5 by a respected wire 22 is connected and whereby from the pressure retaining valve 21 an exit gas line 23 goes off and whereby between the parliamentary group divisor 20 and the pressure retaining valve 21 a N₂-Leitung 33 is inserted; a pure CSI radiator 26, a parliamentary group divisor 27, a pressure retaining valve 28 and a product container 31 whereby the pure CSI radiator 26, which is flowmoderately connected fifth parliamentary group divisors 27 and the pressure retaining valve 28 with one another and whereby the upper range of the pure CSI radiator 26 with the pure CSI column 13 by the ninth wire 17 is connected and whereby from the fifth parliamentary group divisor 27 the tenth wire leads 30 to the product container 31 and whereby from the second pressure retaining valve 28 goes off an exit gas line 29 and whereby between the parliamentary group divisor 27 and the pressure retaining valve 28 a N₂-Leitung 32 is inserted. The CSI generator 5 exhibits appropriately at its lower end a discharge device 34 and is appropriately surrounded by a steam jacket 35. The pure CSI Destillationsblase 12 is appropriately equipped with a heating element 36.

Example

Out of the ClCN vaporizer (1) flowed over the first wire (2) approx. 12,5 kg/h gaseous cyanogen chloride over the drying agent container (molecular sieve dryer) (3) through and by the second wire (4) directly under the fluid levels into the CSI generator (5) were introduced. Over a third wire (11) 17.9 to 24.4 were in-led kg/h the likewise gaseous SO₃ from the oleum distillation column (10) into the CSI generator.

In the CSI generator (5) about 150 l of one was mainly from sulphonyl chloride isocyanate

existing fluid, which was held by the heating loop (35), flowed through by heat distribution medium oil, on a temperature from 120 to 160 DEG C. At the head of the raw CSI column (6) the temperature of the gaseous phase between 90 and 110 DEG C amounted to; the vapors became partial condensed in the raw CSI radiator (7) at temperatures of over 35 DEG C. The condensate arrived over the first parliamentary group divisor (8) and over the fourth wire (9) into the pure CSI column (13) above the second parliamentary group divisor (14).

The pure CSI Destillationsblase (12) was warmed up in such a way with the help of a heating element that the liquid phase contained in it exhibited a temperature from 110 to 130 DEG C. The temperature of the vapor phase at the head of the pure CSI column (13) amounted to 85 to 100 DEG C; the vapors became condensed in the pure CSI radiator (15). The condensate arrived as flyback over the third parliamentary group divisor (16) into the column back.

From the second parliamentary group divisor (14) over the pure CSI wire (17) taken vapors were condensed into the radiator (26). Over the fifth parliamentary group divisor (27) and over the wire (30) the pure CSI flowed into the product container (31) with a density from $d_{20,4} = 1.626 \text{ kg/m}^3$ off.

The remaining vapors from the raw CSI radiator (7) flowed over the wire (24) and out of the pure CSI radiator (15) over the wire (25) into the radiator (19); whereby the radiator temperature was held from 35 to 50 DEG C.

The condensate arrived over the fourth parliamentary group divisor (20) and the wire (22) into the CSI generator (5).

Remaining the not condensable gases left the system over the inert gas barrier (pressure retaining valve) (21) and the wire (23).

The method was accomplished under N₂-Druck up to 0,5 bar positive pressure.


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1. Method to the continuous making of sulphonyl chloride isocyanate by conversion of cyanogen chloride and sulphur trioxide at temperatures from 30 to 160 DEG C, characterised in that one gaseous sulphur trioxide and gaseous cyanogen chloride in a molar ratio of 1, 1 to 1,7:1 in with more chlorsulfonylisocyanathaltiger, at temperatures between 30 to 160 DEG C held, fluid filled reaction zone introduces and to react leaves; that one uses the heat of reaction developing thereby for the operation of the distillation column; that one the vapors in a first cooling zone downstream on a temperature of under 110 DEG C, withdrawing above from the distillation column, cools and condensed down; the fact that one leads back the raw sulphonyl chloride isocyanate resulting thereby partially as flyback into the raw sulphonyl chloride isocyanate column which into the lift zone of a second distillation column downstream leads other part; that one takes the sulphonyl chloride isocyanate out of the second distillation zone between the lift zone and the drift zone and lets in a cooling zone the pure sulphonyl chloride isocyanate condense.

2. Method after claim 1, characterised in that one gaseous sulphur trioxide and gaseous cyanogen chloride in a molar ratio from 1,3 to 1,5:1 into the reaction zone introduces.

3. Method after the claims 1 and 2, characterised in that one the method under inert gas up to a positive pressure of 0,5 bar accomplishes.

4. Verfahren after the claims 1 to 3, characterised in that one above from the second distillation column withdrawing the vapors in a cooling zone downstream on temperatures from 60 to 100 ^{top}DEG C cools down and partial condensed and the condensate than return flow into the second distillation column leads.

5. Method after claim 4, characterised in that one the not condensed vapors from the cooling zone downstream in a further cooling zone downstream under 50 DEG C continues to liquefy

and the condensate into the reaction zone does not introduce.

6. Method after claim 1, characterised in that one the not condensed vapors in the first cooling zone downstream in a further cooling zone downstream under 50 DEG C continues to liquefy and the condensate into the reaction zone does not introduce.

7. Anlage to the lead-through of the method after one of the claims 1 to 5, characterized through, a drying agent container (3), a sulphonyl chloride isocyanate generator (5), a raw sulphonyl chloride isocyanate column (6), a parliamentary group divisor (8), and raw chlorine sulphonyl-isocyanate-coolly (7), whereby on the one hand the drying agent container (3) is led connected by a first wire (2) with the ClCN vaporizer (1) and on the other hand across a second wire (4) to the sulphonyl chloride isocyanate generator (5) under the liquid level and where with on the sulphonyl chloride isocyanate generator (5) the raw sulphonyl chloride isocyanate column (6), the parliamentary group divisor (8) and the raw sulphonyl chloride isocyanate radiator (7) are put on; an oleum distillation zone (10) whereby the upper range of the oleum distillation zone (10) with the sulphonyl chloride isocyanate generator (5) by a third wire (11) is connected; a pure sulphonyl chloride isocyanate distillation blister (12), a pure sulphonyl chloride isocyanate column (13), a lower pure sulphonyl chloride isocyanate parliamentary group divisor (14), an upper parliamentary group divisor (16) and a radiator (15), whereby on the distillation blister (12), the two parliamentary group divisors (14) are put the pure sulphonyl chloride isocyanate column on (13), (16), and the radiator (15), and whereby the upper range of the pure sulphonyl chloride isocyanate column (13) is connected with the parliamentary group divisor (8) by a fourth wire (9) and whereby a fifth wire (18) the pure sulphonyl chloride isocyanate distillation blister (12) with the sulphonyl chloride isocyanate generator (5) and whereby the lower pure sulphonyl chloride isocyanate parliamentary group divisor (14) connects below the intake line (9) is appropriate and whereby of the parliamentary group divisor (14) a product line (17) goes off; a radiator (19), a parliamentary group divisor (20) and a Druckhal teventil (21) whereby the upper range of the radiator (19) is connected by a sixth wire (24) with the upper range of the raw sulphonyl chloride isocyanate radiator (7) and by a sieved wire (25) with the upper range of the radiator (15) and whereby the radiator (19), the fourth parliamentary group divisor (20) and the first pressure retaining valve (21) with one another are flowmoderately connected and whereby the fourth parliamentary group divisor (20) is connected with the sulphonyl chloride isocyanate generator (5) by a respected wire (22) and whereby from the pressure retaining valve (21) an exit gas line (23) goes off and whereby between the parliamentary group divisor (20) and the pressure retaining valve (21) a N2-Leitung (33) is inserted; a pure sulphonyl chloride isocyanate radiator (26), a parliamentary group divisor (27), a pressure retaining valve (28) and a product container (31) whereby the pure sulphonyl chloride isocyanate radiator (26), the fifth parliamentary group divisor (27) and the pressure retaining valve (28) with one another are flowmoderately connected and whereby the upper range of the pure sulphonyl chloride isocyanate radiator (26) with the pure sulphonyl chloride isocyanate column (13) by the ninth wire (17) is connected and whereby from the fifth parliamentary group divisor (27) the tenth wire (30) to the product container (31) and whereby from the second pressure retaining valve (28) an exit gas line (29) leads goes off and whereby between the parliamentary group divisor

(27) and the pressure retaining valve (28) a N₂-Leitung (32) is inserted.

8. Plant according to claim 7, characterised in that the sulphonyl chloride isocyanate generator (5) at its lower end a discharge device (34) exhibits; that the raw sulphonyl chloride isocyanate blister is surrounded by a steam jacket (35).

9. Plant according to claim 7 or 8, characterised in that the pure sulphonyl chloride isocyanate distillation blister (12) with a heating element (36) is equipped.